An “in situ” study of study of quenched Ti-Al system samples during heating

IOP Conf. Series: Materials Science and Engineering **613** (2019) 012007 doi:10.1088/1757-899X/613/1/012007

An “in situ” study of study of quenched Ti-Al system samples during heating

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Abstract. The study of the Ti-Al system samples containing up to 10 wt.% Al water quenched from different temperatures in β-phase region, was carried out in situ during heating on X-ray diffractometer. XRD analysis of quenched samples showed the presence of α’-martensite only. After quenching the increase of Al content in alloys displayed the decrease of the lattice parameters «a» and «c» and the rise of «c/a» ratio, since the lattice parameter «c» dropped slightly in contrast with the lattice parameter «a». Furthermore, Thermo-XRD (T-XRD) analysis represented the anisotropy of the coefficient of thermal expansion (CTE). Besides, T-XRD analysis indicated, that «c/a» ratio of samples Ti-0.5 wt.% Al with the least amount of Al revealed the fall of «c/a» ratio and at the same time samples contained 7 and 8 wt.% Al remained practically the same during heating. Whereas, samples Ti-10 wt.% Al with the largest quantity of Al showed the rise of c/a ratio during heating.

1. Introduction

The objective of this work was the study of the «c/a» parameter changes during the heating compared with room temperature values. Regularities of parameters «c/a» change allow more accurately determine the HCP lattice deformation behavior of α-, pseudo-α- and (α+β)-titanium alloys of different composition with changing deformation temperature, since it directly depends on the alloy composition and, accordingly, on the parameter «c/a» [1, 2].

2. Experiment and Research Methodology

The research material were samples of plates with a thickness of 3 mm made of titanium alloys with different aluminum content. The specimens were water quenched and annealed at 300 º (1 hour). The charge and obtained compositions of the studied alloys are shown in table 1.

Table 1 Chemical composition and quenching temperatures of studied specimens

№	*Al content, weight %*		*Quenching temperature, °C*	*Exposure time, h*
	charge composition	*chemical analysis*		
1	0	0,25	930	0,5
2	7	5,91	1100	0,5
3	8	7,12	1100	0,5
4	10	9,85	1100	0,5

Cutting of samples was carried out on a wire erosion machine ECOCUT. Hot pressing was carried out on a CitersPress-20 press by Struers, PolyFast resin was used. Micrographs were prepared on a LaboPol-5 grinding and polishing machine LaboForce-1 with the sequential use of SiC sanding paper for removing scale and leveling the surface of the sample. Electropolishing of thin sections was carried out in a solution of 20% HClO4 + 80% CH3COOH (glacial acetic acid), at a temperature of 4–8 ° C, current density I = 1–2 A / cm2. The samples were etched in an aqueous solution of nitric and hydrofluoric acids (1 h. HNO3, 1 h. HF, 8 h. H2O).

The study of quenched and annealed samples was carried out by X-ray diffraction analysis (XRD) and Thermo-XRD (TXRD). Both analysis were performed by a BRUCKER D8 X-ray diffractometer equipped electric furnace with a vertical θ-θ-goniometer in the angular range 2θ = 30 ... 120 ° in Kα Cu radiation (λ = 0.154178 nm) in the temperature range 30 ... 290 ° C (temperature step of 65 °). Diffractograms were also obtained after cooling at 30 ºC.

The diffraction patterns were processed using TOPAS software.

3. Results and Discussions

Figure 1 illustrates the coincidence of the position of the experimentally obtained (blue) and reference diffractograms for the α-phase (red). The crystal lattice parameters «a» and «c» were calculated using the TOPAS software. Table 2 represents the results of the calculation of the periods «a», «c», the «c/a» ratio as well as the measurement errors.

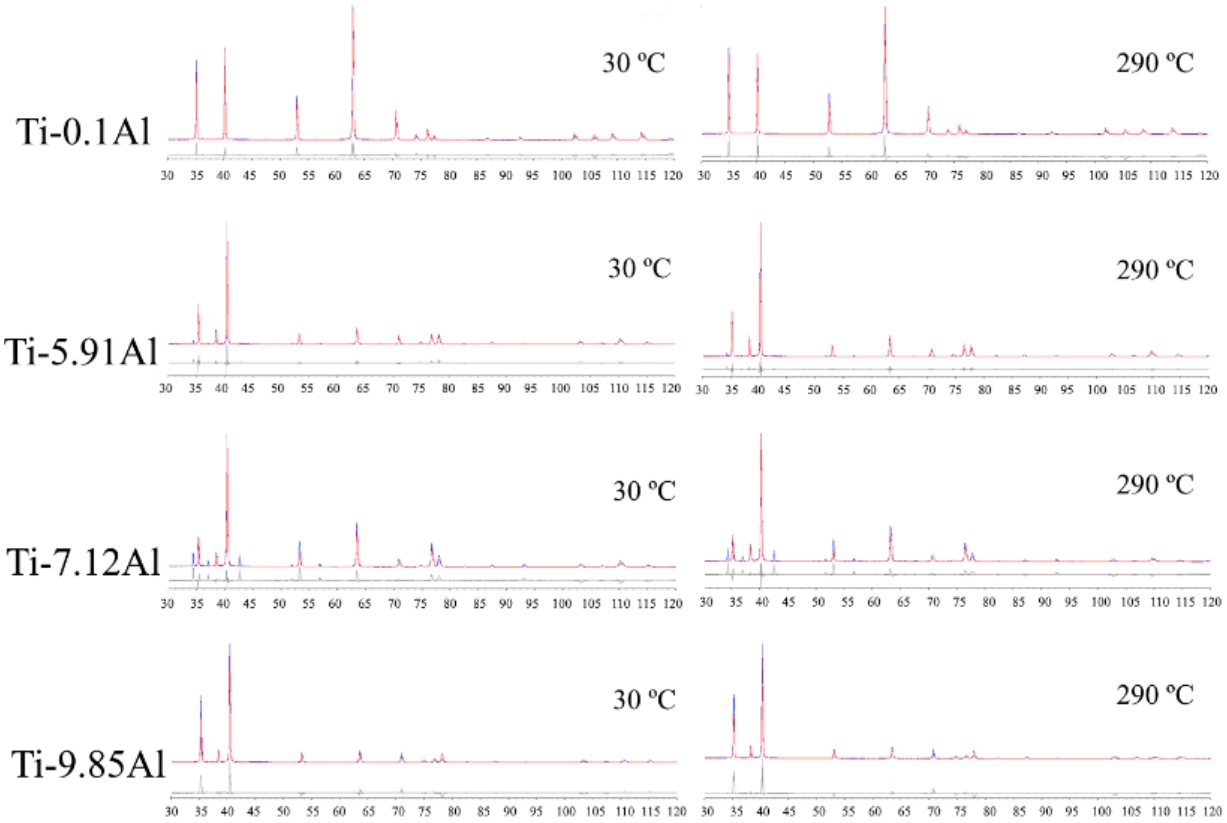

Figure 1. The diffraction patterns taken at the initial and maximal temperatures in the angular range 2θ = 30 ... 120 ° and the second line is the difference between theoretically calculated by the Rietveld method and experimental diffractograms

Table 2 Orthorhombic martensite lattice parameters of WQ and annealed specimens

№	*Al content*		*a, nm·10*	*Δa, nm·10*	*c, nm·10*	*Δc, nm·10*	*c/a*	*Δ(c/a)*
	weight. %	*at. %*						
1	0,25	0,44	2,9501	0,0001	4,6845	0,0002	1,5879	0,0002
2	5,91	10,03	2,932	0,0002	4,6723	0,0004	1,5935	0,0002
3	7,12	11,98	2,929	0,0001	4,6712	0,0002	1,5948	0,0002
4	9,85	16,25	2,9215	0,0002	4,670	0,0003	1,5984	0,0001

A graphic illustration of the obtained values of the periods of the crystal lattice is presented in Figure 2. The resulting graphs clearly show an increase in the periods «a» and «c» depending on temperature, and the pattern obtained is well described by a linear function (coefficient of determination R^2 in all cases > 0.97). The calculation of the periods on the diffraction patterns obtained at 30 ºC before and after cooling showed the same values within the error. A similar experiment for technically pure titanium [3] also revealed the existence of a «c/a» parameter decrease in the temperature range from 25 to 480 ºC. About 6% aluminum alloying leads to temperature stabilization of this parameter, and this ratio begins to increase as the temperature rises to 290 ºC at higher concentrations of aluminum.

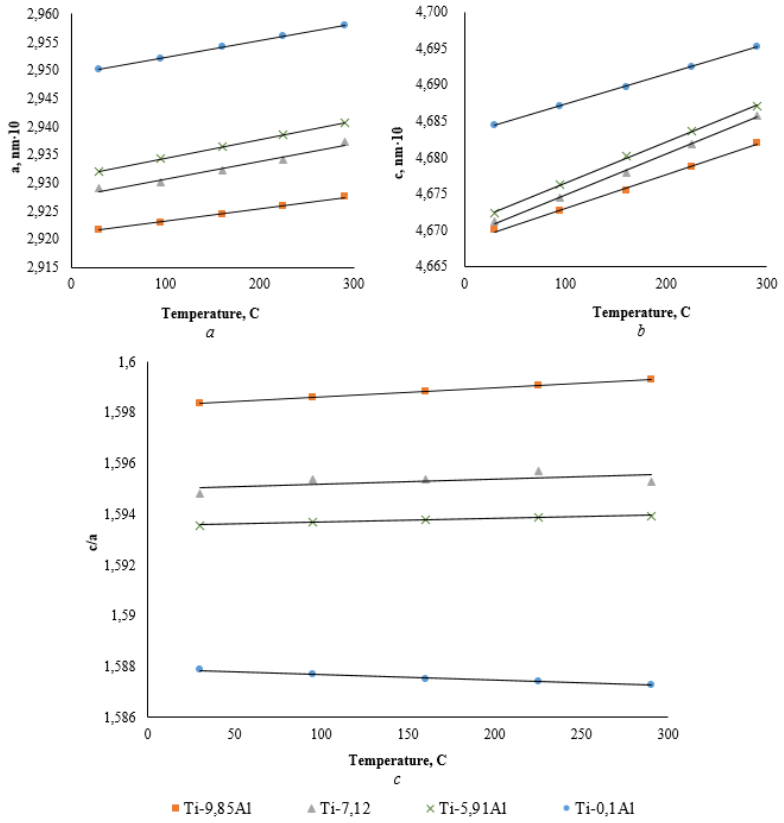

Figure 2. The graphs of the parameters «a» *(a),* «c» *(b)* and «c/a» *(c)* on the aluminum content in the alloys studied during heating in the temperature range of 30 ... 290 ° C

4. Conclusions

The Ti-alloys of the Ti-Al system, the ratio c/a does not remain constant at room and T=290 ºC temperatures. During heating from 30 ºC to 290 ºC of an alloy with a minimum Al content (Ti-0.1Al), the initial value of the ratio decreases to «c/a» = 1.5873. In case of Ti-5.91Al sample, this ratio remained almost the same in the process of heating. In Ti-7.12 Al and Ti-9.85Al alloys the ratio of lattice parameters «c/a» increased.

As a result of the study, data available in the literature on the behavior of α'-HCP martensite lattice alloying with an aluminum and under temperature exposure were confirmed. Also, new regularities were revealed in the change of the ratio of the lattice parameters «c/a» alloying and temperature changed.

Acknowledgement

This article was prepared with the financial support of Competitiveness Enhancement Program – CEP 3.1.1.1-18

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